

A method of producing an unrecrystallized aluminum based thin gauge flat rolled, heat treated product.

Publication number: EP0368005

Publication date: 1990-05-16

Inventor: CHO ALEX

Applicant: ALUMINUM CO OF AMERICA (US)

Classification:

- international: C22C21/10; C22F1/00; C22F1/04; C22F1/053; C22F1/057; C22C21/10; C22F1/00; C22F1/04; C22F1/053; C22F1/057; (IPC1-7): C22C21/10; C22F1/04; C22F1/053

- European: C22C21/10; C22F1/04; C22F1/053; C22F1/057

Application number: EP19890118810 19891010

Priority number(s): US19880256520 19881012; US19880256521 19881012; US19880256840 19881012

Also published as:

JP2194153 (A)
EP0368005 (B1)

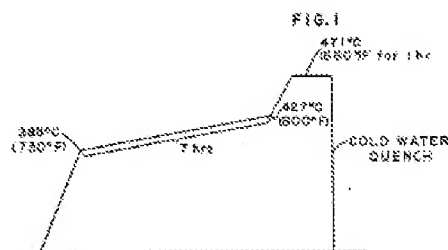
Cited documents:

EP0266741
EP0157600
US4358324
US4486244
WO8000711
more >>

[Report a data error here](#)

Abstract of EP0368005

A treatment of aluminum alloy produces an unrecrystallized Al-Zn-Mg thin gauge flat rolled product having improved levels of strength and fracture toughness. The method comprises providing a body of a Zn-Mg containing aluminum base alloy, working the body to a flat rolled product and then subjecting the product to a ramp anneal followed by solution heat treating, quenching and aging as shown in Fig. 1. The rolled product may be subjected to an isothermal soak before annealing. The working may comprise intermediate heat treating.



Data supplied from the **esp@cenet** database - Worldwide

⑫

EUROPEAN PATENT APPLICATION

⑲ Application number: **89118810.4**

⑤ Int. Cl.⁵: **C22F 1/04, C22F 1/053,**
C22C 21/10

⑳ Date of filing: **10.10.89**

③ Priority: **12.10.88 US 256520**
12.10.88 US 256521
12.10.88 US 256840

④ Date of publication of application:
16.05.90 Bulletin 90/20

⑧ Designated Contracting States:
DE FR GB

⑦ Applicant: **ALUMINUM COMPANY OF**
AMERICA
Alcoa Building
Pittsburgh Pennsylvania(US)

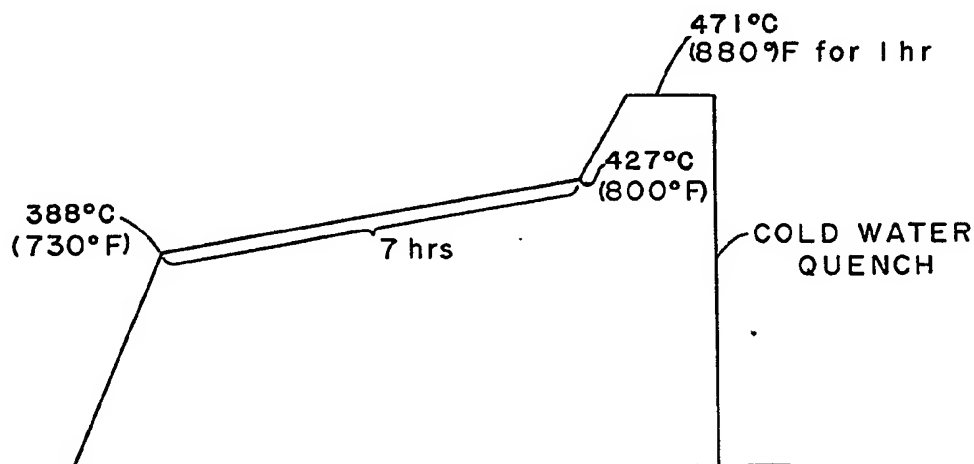
⑦ Inventor: **Cho, Alex**
10025A Palace Court
Richmond Virginia 23233(US)

⑦ Representative: **Baillie, Iain Cameron et al**
c/o Ladas & Parry Isartorplatz 5
D-8000 München 2(DE)

⑤ A method of producing an unrecrystallized aluminum based thin gauge flat rolled, heat treated product.

⑤ A treatment of aluminum alloy produces an unrecrystallized Al-Zn-Mg thin gauge flat rolled product having improved levels of strength and fracture toughness. The method comprises providing a body of a Zn-Mg containing aluminum base alloy, working the body to a flat rolled product and then subjecting the product to a ramp anneal followed by solution heat treating, quenching and aging as shown in Fig. 1. The rolled product may be subjected to an isothermal soak before annealing. The working may comprise intermediate heat treating.

FIG. 1



EP 0 368 005 A1

UNRECRYSTALLIZED ALUMINUM PLATE PRODUCT BY RAMP ANNEALING

This invention relates to heat treatable alloys such as the AA2000, 6000 and 7000 series alloys and more specifically, it relates to thermal or thermal mechanical processing of such alloys to improve strength and fracture toughness in thin plate, for example.

For many years, alloys of the 7000 series have been used for high strength and toughness in aerospace applications. These alloys can be age hardened to very high strengths, for example, in the T6 temper condition. Further, the strengths of these alloys may be increased by increasing solute content. Increasing the strength of these alloys permits designers to reduce the weight of aircraft by reducing thickness of load carrying components such as upper wing skins. Such components must have (and even demand) relatively high fracture toughness as well as high strength to be useful. Several sources indicate that plate having an unrecrystallized structure develops higher toughness than plate having a recrystallized structure. It is well known by those skilled in the art that maintaining the rolling temperature at a high level, typically above about 750° F, allows the aluminum alloy to dynamically recover with a fine subgrain structure, typically about 1 to 2 μ m. This dynamically recovered structure is resistant to recrystallization during subsequent solution heat treatment. However, as the increased strength and toughness allows the use of thinner gauges, prior fabricating techniques and thermal mechanical practices often do not permit production of such products with an unrecrystallized structure because of the tendency for the rolling temperature to fall as the plate thickness is reduced.

Prior art teaches how to achieve recrystallized grain structure but not how to achieve unrecrystallized structure. In the prior art, U.S. Patent 4,092,181 discloses a method of imparting a fine grain recrystallized structure to aluminum alloys having precipitating constituents. The method is provided for imparting a fine grain structure to aluminum alloys which have precipitating constituents. The alloy is first heated to a solid solution temperature to dissolve the precipitating constituents in the alloy. The alloy is then cooled, preferably by water quenching, to below the solution temperature and then overaged to form precipitates by heating it above the precipitation hardening temperature for the alloy but below its solution treating temperature. Strain energy is introduced into the alloy by plastically deforming it at or below the overaging temperature used. The alloy is then subsequently held at a recrystallization temperature so that the new grains are nucleated by the over-

aged precipitates and the development of these grains results in a fine recrystallized grain structure. This structure is useful for imparting superplastic properties but will provide lower toughness than an unrecrystallized structure.

In contrast, the present invention provides improved thermal or thermal mechanical processing techniques which permit the fabrication of flat rolled products, particularly thin gauge plate and sheet 7000 series aluminum alloys having a substantially unrecrystallized structure which imparts to the plate improved combinations of strength and fracture toughness.

In accordance with the invention there is provided an unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin gauge flat rolled product comprised of an aluminum base alloy selected from 2000, 6000 or 7000 series alloys. For 7000 series, the alloy can consist essentially of 1.0 to 12 wt.% Zn, 0.5 to 4.0 wt.% Mg, max. 3.0 wt.% Cu, max. 1.0 wt.% Mn, max. 0.5 wt.% each of Si, Fe, Cr, Ti, Zr, Sc and Hf, the balance aluminum and impurities.

Also, there is provided a method of producing an unrecrystallized, thin gauge flat rolled product which includes hot working a body of the alloy to a thin gauge flat rolled product then subjecting the product to a ramp anneal wherein the annealing temperature is increased with time of anneal. This is followed by solution heat treating, quenching and aging to provide a substantially unrecrystallized product having improved levels of strength and fracture toughness.

Also, there is provided a method of producing an unrecrystallized aluminum base alloy, thin gauge flat rolled product which includes hot working a body of the alloy to a final gauge flat rolled product. This is followed by isothermal soaking the product, ramp annealing, solution heat treating, quenching and aging to provide a substantially unrecrystallized product having improved levels of strength and fracture toughness.

Also, there is provided a method of producing an unrecrystallized Al-Zn-Mg, thin gauge flat rolled product which includes hot working a body of the alloy to a first product. The first product is then reheated, cooled and heat treated before rolling to a thin gauge flat rolled product, e.g., thin gauge plate or sheet. This is followed by solution heat treating, quenching and aging to provide a substantially unrecrystallized product having improved levels of strength and fracture toughness.

Figure 1 is a diagram of a ramp anneal in accordance with the invention.

Figure 2 is a diagram of a ramp anneal in accordance with the invention.

Figure 3 is a schematic representing steps in the process for producing thin gauge unrecrystallized plate in accordance with the invention.

Figure 4 is a schematic representing steps in the process for producing thin gauge unrecrystallized plate in accordance with the invention.

Aluminum based alloys which respond to thermal mechanical processing in accordance with the present invention include the Aluminum Association 7000 series. Such alloys include, for example, 7050, 7150, 7075, 7475, 7049 and 7039.

Typically, these aluminum based alloys contain 1.0 to 12.0 wt.% Zn, 0.5 to 4.0 wt.% Mg, max. 3.0 wt.% Cu, max. 1.0 wt.% Mn, max. 0.5 wt.% each of Si, Fe, Cr, Ti, Zr, Sc and Hf, the balance aluminum, incidental elements and impurities. These alloys may be referred to as Al-Zn-Mg or Al-Zn-Cu-Mg type. Alloys which seem to respond more readily to thermal mechanical processing in accordance with the present invention include higher levels of zinc, preferably 7.0 to 12.0 wt.% Zn with a typical level being 8.0 to 11.0 wt.%. Magnesium at these levels of zinc can range from 0.2 to 3.5, preferably 0.4 to 3.0 wt.%. Also, copper at the higher zinc levels can range from 0.5 to 3.0 wt.%, preferably 1.0 to 3.0 wt.%. These alloying elements may be higher in certain cases, but the resulting alloys can have low fracture toughness. In certain cases, other ranges of alloying elements may be preferred. For example, Zn can be in the range of 7.0 to 9.0 wt.%, Mg 1.5 to 2.5 wt.%, Cu 1.9 to 2.7 wt.%, Zr, 0.08 to 0.14, with impurities such as Fe and Si being less than 0.3 wt.%. The Aluminum Association composition limits encompassing 7050 and 7150 are: 5.7 to 6.9 wt.% Zn, 1.9 to 2.7 wt.% Mg, 1.9 to 2.6 wt.% Cu, 0.05 to 0.15 wt.% Zr, max. 0.12 wt.% Si, max. 0.15 wt.% Fe, max. 0.10 wt.% Mn, max. 0.06 wt.% Ti, max. 0.04 wt.% Cr, the balance aluminum and incidental elements and impurities.

While the AA7000 series aluminum alloys have been described in detail, it will be understood that the invention can be applied to other heat treatable alloys such as the AA2000 and 6000 series aluminum alloys as well as AA8000 alloys which include lithium, e.g., 8090 and 8091. Thus, typical AA2000 series alloys which may be included are AA2024, 2124, 2324, 2219, 2519, 2014, 2618, 2034, 2090 and 2091, and typical of AA6000 series alloys are 6061 and 6013. Products formed from these alloys have oxygen content of less than 0.1 wt.%. Further, the products, e.g., flat rolled products, are substantially free of the as-cast structure.

As well as providing the alloy product with controlled amounts of alloying elements as described herein, it is preferred that the alloy be

prepared according to specific method steps in order to provide the most desirable characteristics of both strength and fracture toughness. Thus, the alloy as described herein can be provided as an ingot or billet for fabrication into a suitable wrought product by casting techniques currently employed in the art for cast products, with continuous casting being preferred. The ingot or billet may be preliminarily worked or shaped to provide suitable stock for subsequent working operations. Prior to the principal working operation, the alloy stock is preferably subjected to homogenization, and preferably at metal temperatures in the range of 850 to 1050° F for a period of time of at least one hour to dissolve soluble elements and to homogenize the internal structure of the metal. A preferred time period is about 20 hours or more in the homogenization temperature range. Normally, the heat up and homogenization treatment does not have to extend for more than 40 hours; however, longer times are not normally detrimental. A time of 20 to 40 hours at the homogenization temperature has been found quite suitable.

In one aspect of the present invention, to produce an unrecrystallized flat rolled product, the ingot may be rolled to a final gauge product. Then, the product is subjected to an annealing treatment wherein annealing temperature is increased with time of anneal and referred to herein as a ramp anneal. In the anneal practice, the starting temperature can be as high as 750° F and then increased with anneal time to temperatures higher than 750° F, e.g. 850° F. With respect to higher starting temperatures, a typical starting temperature is 730° F and the temperature can then be increased with time to about 800° F. When lower ramp anneal temperatures are used, starting temperatures do not usually exceed 550° F, normally 400° F, with a typical starting temperature being in the range of 350 to 450° F and an ending temperature being in the range of 650 to 850° F. Typical ending temperatures are in the range of 750 to 850° F, depending on the alloy composition. In the ramp anneal, the temperature can be increased at a rate of 2 to 100°/hr, and preferably at a rate of 5 to 80°/hr. The time from the beginning to the end of the ramp anneal can range from 3 to about 10 hours, with typical times being in the range of 2 to 8 hours. The ramp anneal can include a series of increases in temperature with a holding time at temperature plateau or series of plateaus. Further, it can include even increases in temperature followed by decreases in temperature until the final ending temperature is reached. Also, there may be even holding plateaus at any one or more temperature level. It will be understood that in some cases, as the anneal temperature gets higher, an independent solution heat treatment may not be necessary

but, instead, is included as part of the ramp anneal, as shown in Figures 1 and 2, or the product may be cooled and a separate solution heat treatment, quench and aging performed.

In a second aspect of the invention, to produce an unrecrystallized product the ingot may be hot rolled directly to final gauge plate or sheet before being subjected to an isothermal soak and then ramp annealed in accordance with the present invention. Thus, to provide a sheet product or plate in accordance with the invention, particularly thin gauge plate product, the product is subjected to a warm temperature or isothermal soak treatment. Thus, the isothermal soak can be carried out at a temperature as low as 250° F but normally higher than 275° F and typically in the range of 300 to 500° F. The soak can be for a time of a few hours, e.g., 3 hours, particularly if the temperature is high and can extend for 24 hours or more. Typically, the soak time extends for 4 to 20 hours. Thereafter, the flat rolled product is subjected to a ramp anneal where the anneal temperature is increased with time of anneal and is carefully controlled until it reaches a higher ending temperature. Preferably, such ending temperatures are in the range of 650 or 700 to 900° F. The starting temperature can be anywhere from about 100° F or even ambient to 750° F in some instances. Typically, the starting temperature will be in the range of 250 to 730° F with preferred starting temperatures being less than 300° F, but normally in the range of 300 to 500° F. From the starting temperature to the ending temperature, the temperature can be increased at a controlled rate, e.g., at a rate of 2 to 125° F/hr and preferably at a rate of 5 to 80° F/hr. The ramp anneal can include a series of increases in temperature with a holding time at temperature plateau or series of plateaus. Further, it can include even increases in temperature followed by decreases in temperature until the final ending temperature is reached. Also, there may be even holding plateaus at any one or more temperature level. It will be understood that in some cases, as the anneal temperature gets higher, an independent solution heat treatment may not be necessary but, instead, is included as part of the ramp anneal, as shown in fig. 3, or the product may be cooled and a separate solution heat treatment, quench and aging performed. The time from the beginning of the ramp anneal to the ending temperature can be as short as two hours or even less to as long as 20 hours or more. Time periods in the range of 3 to 10 hours have been quite suitable with time periods of 4 to 8 hours being found to be useful.

Use of isothermal soak and ramp anneal as disclosed here has been found to be quite beneficial because this process appears to be somewhat insensitive to practices employed to work the ingot.

In certain alloys, to obtain an unrecrystallized product, it may be desirable to combine these processes. That is, the ramp anneal may be used in addition to precipitation heat treating intermediate the working steps, and such combination is contemplated within the purview of the invention.

Thus, an unrecrystallized thin gauge plate or sheet product may be produced in a third aspect of the present invention.

By unrecrystallized is meant the absence of well-developed grains and the presence of a highly worked structure containing recovered subgrain and retaining as-worked crystallographic texture, i.e., at least 60% of the plate or sheet is free of well-developed grains or retains the as-worked texture. In this process, the thermomechanical steps should be carefully controlled. Thus, after homogenization of the ingot and hot rolling to a slab dimension, the hot rolling performed at temperatures in the range of 500 to 900° F, the slab is reheated typically to a temperature in the range of 650 to 900° F and preferably 650 or 700 to 800° F (depending upon composition), for purposes of dissolving or partially dissolving particles that precipitated during the preceding thermal mechanical operation. Reheating can be carried out in a time as short as $\frac{1}{4}$, or $\frac{1}{2}$ hour at temperature, and can extend for 4 hours or more. However, the longer times are not normally necessary. Then, the slab is cooled at a rate sufficient to retain dissolved elements in solution. Preferably, the slab is cold water quenched or rapidly cooled. Thereafter, the slab is subjected to an elevated temperature precipitation heat treatment to precipitate particles in a controlled manner. The precipitation heat treatment can be carried out at a temperature in the range of 200 to 550° F, preferably 350 to 500° F, with typical temperatures being 400 to 500° F. Precipitation heat treatment times at this temperature can range from 5 to 20 hours or longer, and times of from 9 to 15 hours can be quite suitable. After the precipitation heat treatment, the slab is worked or rolled to thin gauge plate or to sheet stock. Thin gauge plate contemplates having a thickness of at least 0.125, typically 0.25 inch or more. The thickness can extend to 0.5 inch or more, for example, 0.75 or 1.0 or even 1.25 inch.

While the slab may be cold rolled at these temperatures, it is preferred that the slab be rolled to final gauge, e.g., thin gauge plate or sheet, using warm rolling practices. Thus, preferably, warm rolling is performed at a temperature of not greater than 550° F. Further, preferably, the temperature at which warm rolling begins is not less than 200° F. Typically, the warm rolling can begin at the precipitation heat treatment temperature. Preferably, the warm rolling temperature should not exceed the precipitation heat treatment temperature. Such tem-

peratures are in the range of about 350 to 500° F. This warm rolling practice contrasts with the prior art which teaches that rolling temperatures should be significantly higher, typically above about 750° F.

Optionally, the plate or sheet product is subjected to a solution heat treatment and then cooled, for example, by cold water quenching.

The solution heat treatment is preferably accomplished at a temperature in the range of 800 to 1050° F and unrecrystallized grain structure is produced. Generally, for sheet gauge typical times at temperatures can be relatively short, e.g., 5 minutes or less can be adequate. For thin gauge plate 0.5 inch, the time at temperature can be $\frac{1}{4}$ to 5 hours, typically 2 hours.

To further provide for the desired strength and fracture toughness necessary to the final product and to the operations in forming that product, the product should be rapidly quenched to prevent or minimize uncontrolled precipitation of strengthening phases. Thus, it is preferred in the practice of the present invention that the quenching rate be at least 100° F per second from solution temperature to a temperature of about 200° F or lower. A preferred quenching rate is at least 200° F per second in the temperature range of 900° F or more to 200° F or less. After the metal has reached a temperature of about 200° F, it may then be air cooled.

After the alloy product of the present invention has been quenched, it may be subjected to a subsequent aging operation to provide the combination of fracture toughness and strength which are so highly desired in aircraft members. Artificial aging can be accomplished by subjecting the sheet or plate or shaped product to a temperature in the range of 150 to 400° F for a sufficient period of time to further increase the yield strength. Some compositions of the alloy product are capable of being artificially aged to a yield strength as high as 100 ksi. However, the useful strengths are in the range of 70 to 90 ksi and corresponding fracture toughnesses are in the range of 20 to 50 ksi/in. Preferably, artificial aging is accomplished by subjecting the alloy product to a temperature in the range of 275 to 375° F for a period of at least 30 minutes. A suitable aging practice contemplates a treatment of about 8 to 24 hours at a temperature of about 325° F. Further, it will be noted that the alloy product in accordance with the present invention may be subjected to any of the typical overaging or underaging treatments well known in the art, including natural aging. However, it is presently believed that natural aging provides the least benefit. Also, while reference has been made herein to single aging steps, multiple aging steps, such as two or three aging steps, are contemplated and

stretching or its equivalent working may be used prior to or even after part of such multiple aging steps.

While the invention has been described with respect to sheet and plate, it will be appreciated that its application is not necessarily limited thereto. That is, the process can be applied to extrusions and forgings having alloy compositions referred to herein or responsive to these treatments. In contrast to rolling, for extrusion purposes, it is not difficult to keep the ingot hot, but it is uneconomical to do so because of the slow extruding rates. Consequently, extrusions typically have a recrystallized structure. To provide an unrecrystallized extrusion in accordance with the invention, the process would include two or more extruding steps. That is, after achieving an ingot temperature of about 700 to 800°, the ingot is extruded to an intermediate cross-sectional area, e.g., to reduce the area 75%. Thereafter, the partially extruded material is subjected to a reheating step, for example, under the same conditions as referred to herein with respect to slab. Also, it is cooled and subjected to an elevated precipitation treatment as referred to for slab, for example. Thereafter, the partial extrusion is further worked or extruded to product form preferably utilizing warm temperatures, for example, under the same conditions referred to for slab being rolled to final gauge. Thereafter, the extrusion may be solution heat treated, quenched and aged to produce an unrecrystallized aluminum alloy extrusion. Because the steps to form forgings are often repeated, the forging operation may be carried out incorporating the procedures set forth for the flat rolled product to produce an unrecrystallized aluminum alloy forged product. It will be appreciated that the rolling, extruding or forging steps may be combined to produce an unrecrystallized product.

This practice of ramp annealing is suitable for use in many applications. That is, it may be used quite successfully regardless of the previous thermomechanical practices. For example, it has been used on thin gauge plate where the slab was reheated, quenched, heat treated and warm rolled to a plate product, described earlier herein, to produce a thoroughly or completely unrecrystallized product (see Example 3).

Example 1

An aluminum alloy consisting essentially of, by weight percent, 10 Zn, 1.8 Mg, 1.5 Cu and 0.12 Zr, the balance essentially aluminum and impurities was cast into an ingot suitable for rolling. The ingot was homogenized and hot rolled at about 800° F to

a 1.5 inch thick slab. The slab was cut into several pieces which were heated to temperatures of 750 to 880° F and then hot rolled starting at about 750° F to 0.3 inch thick plate. Samples were given a ramp anneal starting at a temperature of 730° F and ending at 800° F, with a heat-up rate of about 10° F/hr. After annealing, these samples, along with unannealed samples, were heated to 880° F and solution heat treated at this temperature for 1 hour and then cold water quenched, as shown in Figure 1. Examination of the microstructure revealed that the degree of recrystallization of the ramp annealed samples was significantly reduced compared to the microstructure of samples which were not annealed in this manner.

Example 2

Samples of 0.3 inch plate having the composition and prepared as in Example 1 were subjected to a ramp anneal starting at a temperature of 400 and ending at a temperature of 800° F, the increase in temperature being performed in 4 hours, as shown in Figure 2. These samples were solution heat treated as in Example 1. Examination of the microstructure showed a basically unrecrystallized grain structure.

Example 3

This sample (0.3 inch plate) had the same composition and treated as in Example 2 except that prior to hot rolling to 0.3 inch thick plate, the sample was reheated to 750° F for about 0.5 hours, cold water quenched and then precipitation heat treated at 400° F for 12 hours and hot rolled to 0.3 inch thick plate starting at a temperature of 400° F. The microstructure of this sample revealed a completely unrecrystallized grain structure.

Example 4

An aluminum alloy consisting essentially of, by weight percent, 10 Zn, 1.8 Mg, 1.5 Cu and 0.12 Zr, the balance essentially aluminum and impurities, was cast into an ingot suitable for rolling. The ingot was homogenized and rolled to a 1.5 inch thick slab. The slab was cut into several pieces which were subjected to anneals at temperatures of 750 to 880° F and then hot rolled to 0.3 inch plate. Thereafter, the 0.3 inch plate was isothermal soaked for 16 hours at 400° F and then subjected

to a ramp anneal starting at 400° F and ending at 800° F, increase in temperature being made in 4 hours. The 0.3 inch plate was then solution heat treated at 880° F for 1 hour followed by a cold water quench. Examination of the microstructure showed unrecrystallized grain structures which demonstrates the effectiveness of isothermal soaking and ramp annealing in preventing recrystallization.

Example 5

An aluminum alloy having a nominal weight percent of 10 Zn, 1.8 Mg, 1.5 Cu and 0.12 Zr, the balance essentially aluminum and impurities, was cast into an ingot suitable for rolling. The ingot was homogenized and then hot rolled at about 800° F to a 1.5 inch thick slab. Thereafter, the slab was annealed for 30 minutes at 750° F and cold water quenched. The slab was then precipitation heat treated for 12 hours at 400° F. Thereafter, the slab was rolled at about 400° F to 0.3 inch thick plate and then solution heat treated at 880° F for 1 hour and cold water quenched. Examination revealed that the microstructure was substantially an unrecrystallized microstructure. By comparison, identical samples which were not aged, but hot rolled to 0.3 inch plate immediately after annealing at 750° F showed a high degree of recrystallization. Thus, it will be seen that thermomechanical processing in accordance with the subject invention can produce an unrecrystallized thin gauge plate or sheet product in the Al-Zn-Mg or Al-Zn-Mg-Cu type aluminum alloys.

Claims

1. A method of producing an unrecrystallized aluminum based thin gauge flat rolled, heat treated product having improved levels of strength and fracture toughness, the method comprising the steps of:
 - (a) providing a body of a aluminum base heat treatable alloy;
 - (b) working the body to a wrought product;
 - (c) subjecting said product to a ramp anneal;
 - (d) solution heat treating, quenching and aging said final gauge flat rolled product to provide a substantially unrecrystallized product having improved levels of strength and fracture toughness.
2. A method according to claim 1, wherein the anneal temperature is selected from one of the following:
 - (1) the anneal starts at a temperature of not

greater than 750 °F ;

(2) the anneal starts at a temperature of not greater than 400 °F ;

(3) the anneal ends at a temperature in the range of 650 to 850 °F ;

(4) the anneal starts at a temperature in the range of 350 to 450 °F and is increased to a temperature in the range of 750 to 850 °F in a period of about 2 to 8 hours; and/or in the anneal the temperature is increased at a rate of 2 °/hr to 100 °/hr.

3. A method according to claim 1, wherein before subjecting said product to said ramp anneal, said product is subjected to an isothermal soak.

4. A method according to claim 1, wherein said working comprises the following steps

(1) hot working the body to a first wrought product;

(2) reheating said first wrought product;

(3) cooling said first wrought product;

(4) heat treating said first wrought product;

(5) further working said first wrought product to produce a second wrought product.

5. A method of producing an unrecrystallized aluminum base, wrought alloy product having improved levels of strength and fracture toughness, the method comprising the steps of:

(a) providing a body of an aluminum base heat treatable alloy;

(b) working the body to a wrought product;

(c) subjecting said product to an isothermal soak;

(d) then subjecting said product to a ramp anneal wherein the annealing temperature is increased during time of anneal; and

(e) solution heat treating, quenching and aging said product to provide a substantially unrecrystallized wrought product having improved levels of strength and fracture toughness.

6. A method according to claim 5, wherein the ramp anneal temperature is selected from one of the following:

(1) the ending temperature is 650 to 900 °F ;

or

(2) the starting temperature is less than 300 °F and the ending temperature is 700 to 900 °F; and/or the temperature is increased at 2 to 125 °F per hour, preferably 5 to 80 °F per hour.

7. A method according to claim 5, wherein the isothermal soak is at a temperature in the range of 300 to 500 °F, and/or the duration of said soak is selected from:

(1) at least 3 hours

(2) at least 4 hours;

(3) a period in the range of 4 to 24 hours.

8. A method of producing an unrecrystallized, wrought aluminum base, heat treated product having improved levels of strength and fracture tough-

ness, the method comprising the steps of:

(a) providing a body of an aluminum base, heat treatable alloy;

(b) hot working the body to a first wrought product;

(c) reheating said first wrought product;

(d) cooling said first wrought product;

(e) heat treating said first wrought product;

(f) further working said first wrought product to produce a second wrought product; and

(g) solution heat treating, quenching and aging said second wrought product to provide a substantially unrecrystallized product having improved levels of strength and fracture toughness.

9. A method according to claim 8, wherein the temperature of said reheating is selected from one of the following:

(1) a temperature of 500 to 900 °F;

(2) a temperature of 650 to 800 °F;

(3) a temperature of 700 to 800 °F; and/or the duration of said reheating is for a time of least 1/4 hour, preferably from 1/4 to 4 hours.

10. A method according to claim 8, wherein the heat treating of said first wrought product is performed at a temperature selected from one of the following:

(1) from 200 to 550 °F;

(2) from 350 to 500 °F;

(3) from 400 to 500 °F and/or the duration of said heat treating is from 5 to 20 hours, preferably 9 to 15 hours.

11. A method according to any one of claims 1 to 10, wherein the alloy contains 1.0 to 12 wt.% Zn, 0.5 to 4.0 wt.% Mg, max. 3.0 wt.% Cu, max. 1.0 wt.% Mn, max. 0.5 wt.% each of Si, Fe, Cr, Ti, Zr, Sc and Hf, the balance aluminum and impurities.

12. A method according to any one of claims 1 to 10, wherein the alloy contains 7.0 to 9.0 wt.% Zn, 1.5 to 2.5 wt.% Mg, 1.9 to 2.7 wt.% Cu, 0.08 to 0.14 wt.% Zr, max. 0.5 wt.% each of Si, Fe, Cr, Ti, Zr, Sc and Hf, the balance aluminum and impurities.

13. An unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin flat rolled product comprised of aluminum base alloy consisting essentially of 7.0 to 9.0 wt.% Zn, 1.5 to 2.5 wt.% Mg, 1.9 to 2.7 wt.% Cu, 0.08 to 0.14 wt.% Zr, max. 0.12 wt.% Si, max. 0.15 wt.% Fe, max. 0.10 wt.% Mn, max. 0.06 wt.% Ti, max. 0.04 wt.% Cr, the balance aluminum and incidental elements and impurities, the product having a thickness in the range of 0.1 to 0.75 inch.

14. An unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin flat rolled product comprised of aluminum base alloy consisting essentially of 5.7 to 6.9 wt.% Zn, 1.9 to 2.7

wt.% Mg, 1.9 to 2.6 wt.% Cu, 0.05 to 0.15 wt.% Zr, max. 0.12 wt.% Si, max. 0.15 wt.% Fe, max. 0.10 wt.% Mn, max. 0.06 wt.% Ti, max. 0.04 wt.% Cr, the balance aluminum and incidental elements and impurities, the product having a thickness in the range of 0.1 to 0.75 inch. 5

15. An unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin flat rolled product comprised of aluminum base alloy consisting essentially of 5.7 to 6.9 wt.% Zn, 1.9 to 2.7 wt.% Mg, 1.9 to 2.6 wt.% Cu, 0.05 to 0.15 wt.% Zr, max. 0.12 wt.% Si, max. 0.15 wt.% Fe, max. 0.10 wt.% Mn, max. 0.06 wt.% Ti, max. 0.04 wt.% Cr, the balance aluminum and incidental elements and impurities, the product being substantially free of as cast structure and having a thickness in the range of 0.25 to 0.5 inch. 10 15

16. An unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin flat rolled product comprised of aluminum base alloy consisting essentially of 1.0 to 12 wt.% Zn, 0.5 to 4.0 wt.% Mg, max. 3.0 wt.% Cu, max. 1.0 wt.% Mn, max. 0.5 wt.% each of Si, Fe, Cr, Ti, Zr, Sc and Hf, less than 0.1 wt.% O₂, the balance aluminum and incidental elements and impurities, the product being substantially free of as cast structure and having a thickness in the range of 0.25 to 0.5 inch. 20 25

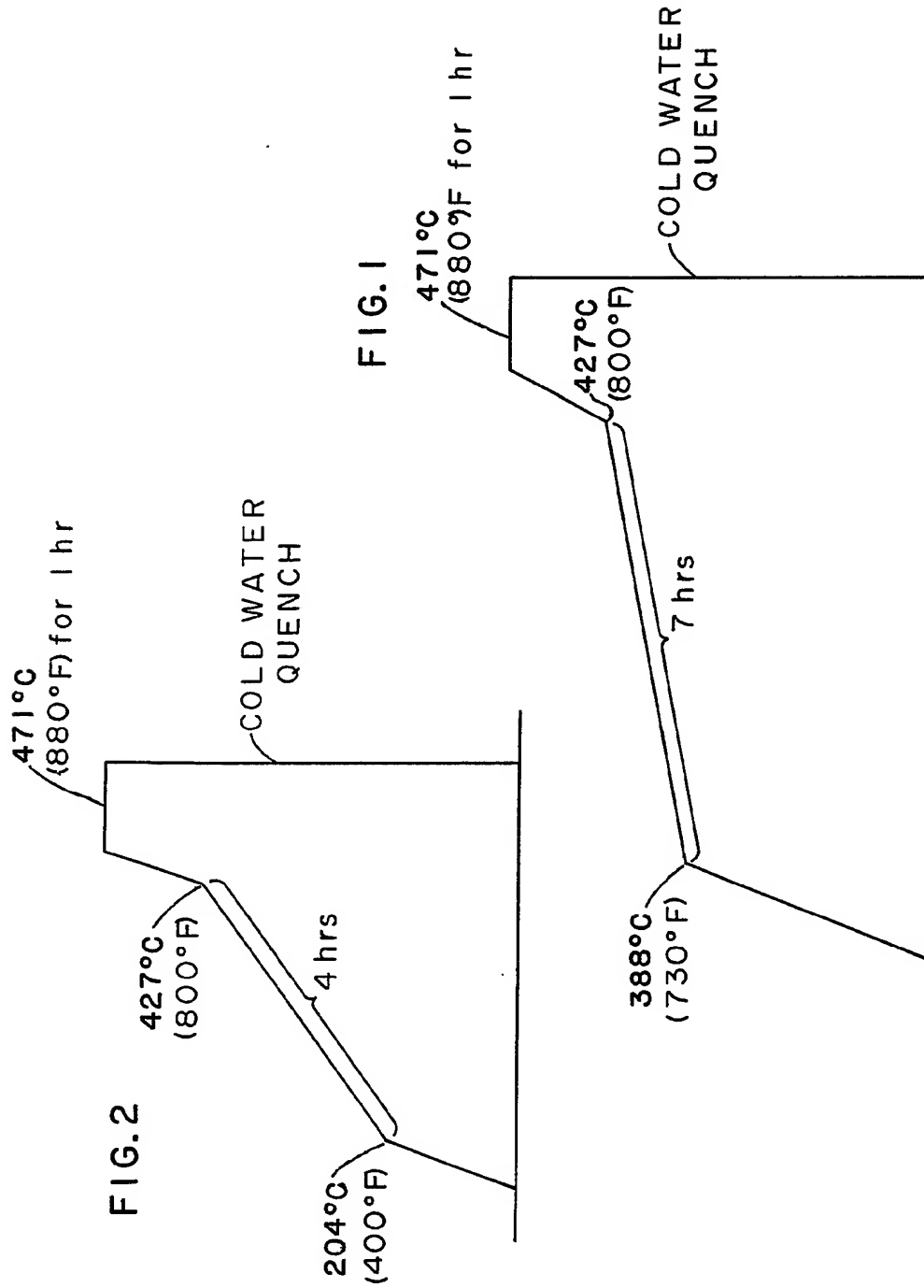
17. An unrecrystallized thin gauge flat rolled product suitable for fabricating into aircraft structural members, the unrecrystallized thin flat rolled product comprised of aluminum base alloy consisting essentially of 7.0 to 9.0 wt.% Zn, 1.5 to 2.5 wt.% Mg, 1.9 to 2.7 wt.% Cu, 0.08 to 0.14 wt.% Zr, max. 0.12 wt.% Si, max. 0.15 wt.% Fe, max. 0.10 wt.% Mn, max. 0.06 wt.% Ti, max. 0.04 wt.% Cr, the balance aluminum and incidental elements and impurities, the product being substantially free of as cast structure and having a thickness in the range of 0.25 to 0.5 inch. 30 35 40

45

50

55

Neu eingereicht / Newly filed
Nouvellement déposé



Not a carbon / New
L'engendrement d'un

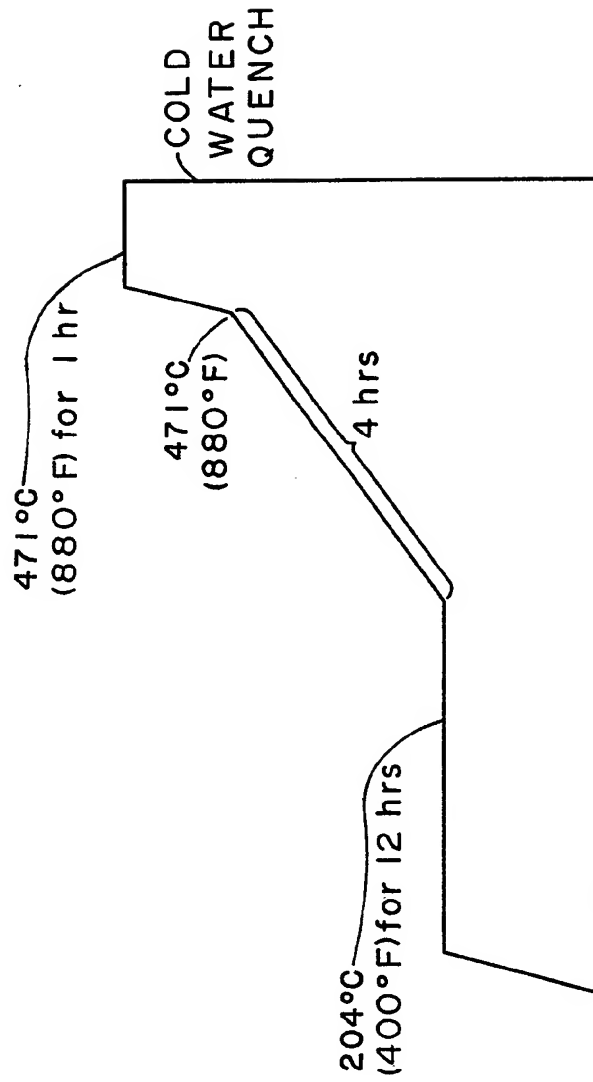


FIG. 3

Neu eingereicht / N
Nouvellement d

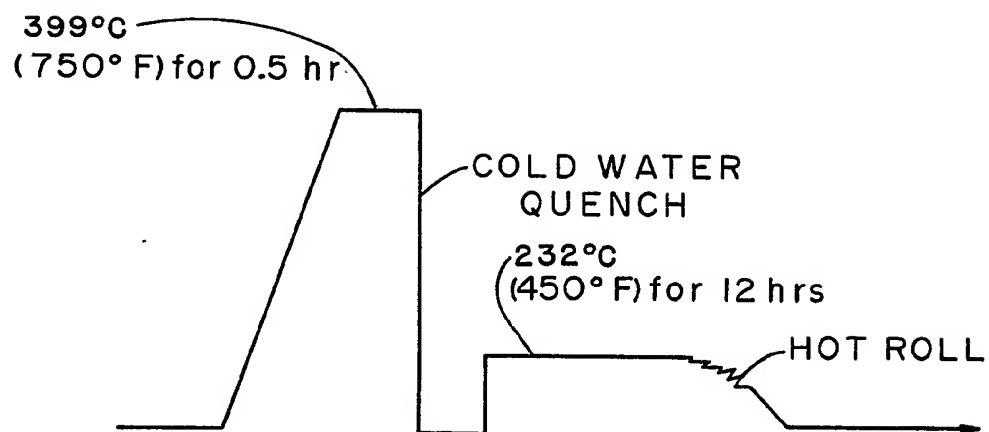


FIG. 4



DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 5)
X	EP-A-0 266 741 (ALUMINIUM CO. OF AMERICA) * Claims 1-4; page 4, lines 45-58 *	1,2	C 22 F 1/04 C 22 F 1/053 C 22 C 21/10
Y	---	11,12	
A	EP-A-0 157 600 (ALUMINIUM CO. OF AMERICA) * Claims 1,6; page 8, line 17 - page 10, line 13 *	1-10	
A	---		
A	US-A-4 358 324 (M.W. MAHONEY et al.) * Claims 1-3 *	1-7,11,12	
A	---		
A	US-A-4 486 244 (B.R. WARD et al.) * Claims 1-4 *	8-10	
X	---		
X	WO-A-8 000 711 (THE BOEING CO.) * Claims 1,6,7; page 1, line 23 - page 2, line 2; page 4, lines 17-23; page 8, lines 4-14 *	14-16	
Y	---	11,13,17	TECHNICAL FIELDS SEARCHED (Int. Cl. 5)
Y	FR-A-2 518 579 (ALUMINIUM CO. OF AMERICA) * Claims 1,6,7; page 5, lines 26-30 *	12,13,17	C 22 F C 22 C
A	---		
A	FR-A-2 113 037 (O. FUCHS) * Claim 1 *	11-17	

The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 30-11-1989	Examiner GREGG N.R.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			